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# EFFECT OF IRRADIATION TEMPERATURE ON NEUTRON-INDUCED CHANGES IN NOTCH DUCTILITY OF PRESSURE-VESSEL STEELS

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## ABSTRACT

In an effort to understand more fully the effects of elevated irradiation temperatures upon the shift (increase) in the ductile-to-brittle transition temperature of two pressure-vessel steels, an experimental assembly containing four groups of specimens of these steels has been irradiated in the core of the Oak Ridge Low Intensity Test Reactor. Through the careful design of each unit and by manipulation of the outer experimental containment sheath by air pressure, four different irradiation temperatures,  $260^{\circ}$ ,  $400^{\circ}$ ,  $450^{\circ}$ , and  $550^{\circ}$  F, were maintained for a one-month irradiation period.

Post-irradiation evaluation of specimens indicated no significant temperature effect for the materials irradiated at less than  $450^{\circ}$  F, the shift in the ductile-to-brittle transition temperature being about the same for each of the three irradiation temperatures; however, there was a significant effect as a result of irradiation at  $550^{\circ}$  F, the shift being roughly one-hundred degrees less. The transition-temperature shifts for materials irradiated at less than  $450^{\circ}$  F were in good agreement with data from nineteen earlier experiments in which the specimen temperatures during irradiation were less than  $200^{\circ}$  F. The shift increases linearly with the logarithm of neutron flux dosage, with data for irradiation below  $450^{\circ}$  F falling along a single line; the data for materials irradiated at  $550^{\circ}$  F fall on a line displaced toward lower transition-temperature shifts.

## PROBLEM STATUS

This report covers preliminary studies of comparison effects of elevated temperatures and neutron irradiation of steels; work on this and other phases of the problem are continuing.

## AUTHORIZATION

NRL Problem M01-14  
Projects SR 007-01-01, Task 0858  
and RR 007-01-42-5409  
AEC(AT49-5)-1907

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## EFFECT OF IRRADIATION TEMPERATURE ON NEUTRON-INDUCED CHANGES IN NOTCH DUCTILITY OF PRESSURE-VESSEL STEELS

### INTRODUCTION

A large amount of the reported data on the effects of neutron radiation has been obtained for thermal environments dissimilar to those present at the pressure-vessel walls of operating pressurized-water nuclear power plants (1-3)\*. In fact, the majority of materials irradiation experiments have been conducted at temperatures under 400° F, whereas, most pressurized-water reactors operate at temperatures between 430° and 550° F.

The results of an earlier investigation have shown that exposure temperature is an important variable in radiation effects studies. The shift in the ductile-to-brittle transition of the steels investigated was significantly less with irradiation temperatures in the 550° to 600° F range as compared to that observed with temperatures under 200° F. The determination of whether neutron-induced changes to steel are progressively reduced with increased temperature (above 200° F), or whether a critical temperature must be exceeded for the initiation of this phenomenon, is therefore of great importance in determining the influence of elevated temperatures during irradiation of steel.

Although an insight into the effects of elevated temperatures on the neutron-induced changes in notch ductility of steels could be obtained with a series of experiments conducted in the same reactor facility, an accurate evaluation of properties after irradiation is possible only if the nuclear environment is identical in each experiment with respect to reactor fuel loading, reactor operating time, power level, etc. To establish a basis for the direct comparison of results, an experimental assembly was designed to provide a series of uniform specimen temperatures simultaneously during irradiation in a vacant fuel element position in the Oak Ridge National Laboratory Low Intensity Test Reactor (LITR). With this assembly, specimens of two commonly used pressure-vessel steels have been exposed at three temperatures within the normal reactor operating range and at one temperature considerably below this range. Although a one-month irradiation period was used, the duration of the reactor exposure is not dictated by the assembly design.

### MATERIALS

The steels obtained for this investigation, ASTM A212 Grade B and A302 Grade B, were supplied by commercial sources and are representative of heat-treated pressure-vessel materials. Chemical analyses and detailed information on the metallurgical histories of the individual materials are presented in the appendix. The full-size Charpy V-notch specimens and the 0.180-in. tensile specimens† used for the investigations were taken from the one-quarter thickness location.

\* These references are only examples of available literature.

† For later investigations on tensile properties.

## REACTOR FACILITY

The experimental facility of the LITR considered appropriate for the multitemperature irradiation experiment was the vacant C-18 fuel element position. Previous NRL experience with this facility indicated that a relatively uniform neutron environment exists over a distance of about 12 inches near the fuel centerline. Figure 1(a) indicates schematically the position of the C-18 facility in the LITR. The relative location of the experimental assembly to the main fuel-loaded section of the reactor core is shown in Fig. 1(b). Average instantaneous neutron flux values ( $>1.0$  Mev) were determined with sulfur and nickel neutron detectors. The flux level on the core side of the assembly was  $2.92 \times 10^{12}$  n/cm<sup>2</sup>-sec and on the opposite side was  $2.15 \times 10^{12}$  n/cm<sup>2</sup>-sec. This flux variation was known from previous experiments (4). The A212B specimens were positioned on the side of the assembly adjacent to the main fuel core and the A302B specimens were aligned adjacent to the fuel element on the opposite side.

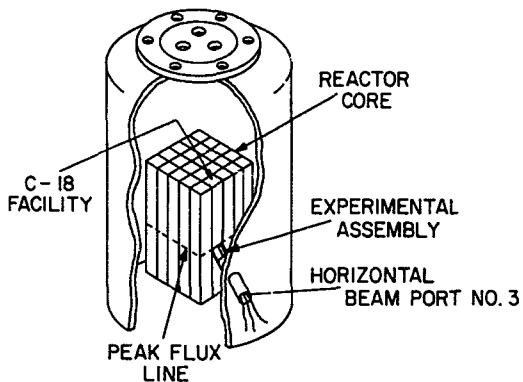
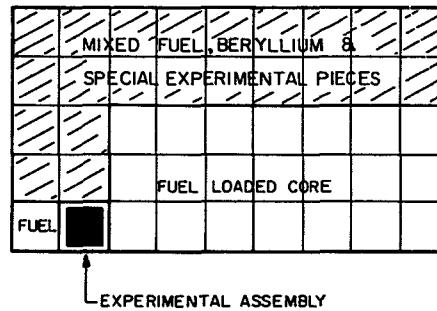


Fig. 1(a) - Schematic sketch showing experiment position in the Oak Ridge National Laboratory Low Intensity Test Reactor

Fig. 1(b) - Irradiation-assembly location in LITR C-18 in relation to reactor core (schematic)



## EXPERIMENTAL ASSEMBLY

In planning the multitemperature assembly, four major design requirements were envisioned. First, the assembly would be of square cross section to fit into the existing dummy fuel element piece and, at the same time, provide a maximum number of specimens in the limited space available. Second, this relatively large number of specimens per assembly section was to serve as a self-heater in the high gamma flux. Third, intimate contact between specimens in each section would be required to minimize transverse thermal gradients. Fourth, temperature control was to be obtained by controlling the rate of heat removal from each section to the coolant water.

Figures 2 and 3 show the major features of the final assembly design. The complete assembly was 10 in. long and consisted of four individual units positioned along its length. Figure 2 shows the individual units before assembly and encapsulation in an overall envelope of 0.010-in.-thick stainless steel as shown in Fig. 3. The external aluminum framework of each unit was incorporated to satisfy the third and fourth design requirements listed above. The framework held the specimens very tightly together and served as the primary heat transfer path from each unit to the stainless steel sheath. In addition to this "built-in" control of unit temperatures, a second mode of control was used for final temperature adjustments during irradiation. Since the stainless steel sheath was flexible, an externally controlled air pressurization within the assembly served to interrupt contact of the sheath with the aluminum framework at certain points, thereby providing a sensitive means of temperature control. Thus, the variations in internal unit construction and in the outer aluminum surface area provided the fixed factors in heat transfer. These, coupled with the feature of a flexible outer sheath, provided the desired variation in temperature between units.

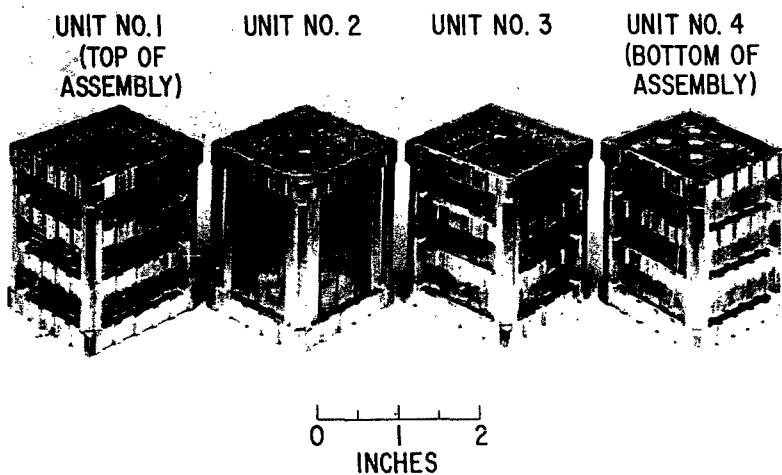


Fig. 2 - The four individual units which combine to form the irradiation assembly, showing variations in internal loading and in outer aluminum framework

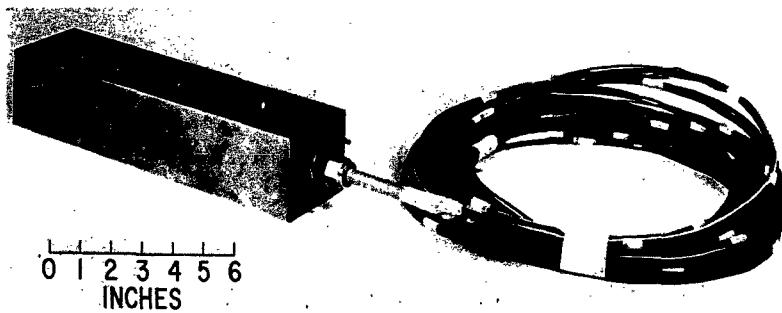


Fig. 3 - Irradiation assembly before enclosure of leads in stainless steel tubing

To provide a record of the irradiation temperature of each unit and to generate a signal for the external temperature control equipment, two thermocouples were attached to specimens in each unit at selected points to detect the temperature extremes in that unit. Thermocouple wires were led to the top of the assembly through a drilled Charpy specimen in the center of each unit.

Neutron flux monitors were also enclosed in drilled Charpy specimens in unit 3 and were placed in holes in the central steel spacing piece of unit 4 (Fig. 2). In addition, nickel-wire flux monitors were situated in the four corners of unit 2.

After the units had been assembled, the outer stainless steel sheath was very carefully formed and welded around the units to obtain the desired contact with the aluminum frames.

Since the assembly was the first experimental model of this type to be irradiated, a 1/16-in. stainless steel tube was also included to provide a helium atmosphere rather than air, if desired. This helium line as well as the thermocouple leads were enclosed in a 3/8-in. stainless steel tube which penetrated the reactor's top access plug. In addition to shielding the thermocouple leads from the reactor coolant water, the 3/8-in. tube provided a connection to the off-gas plenum system and to the assembly pressurization circuit at the temperature control console.

#### ASSEMBLY IRRADIATION

The physical orientation of the experiment in the reactor and the instrumentation of the control console utilized are shown schematically in Fig. 4. The assembly temperature control system is completely flexible in that pressurization by either air or helium, or, the creation of a partial vacuum in the assembly, can be accomplished. No provisions were made for externally controlling the heat supply to the assembly since the gamma heating in the C-18 facility was considered more than sufficient to meet the desired experimental objectives. The elimination of an externally controlled heat source also insured that assembly temperatures would drop to ambient reactor coolant temperatures during shutdown periods. The possibility of an interim specimen annealing treatment occurring during these periods was therefore successfully eliminated.

Assembly unit temperatures were carefully monitored during the initial reactor startup to determine the optimum method of temperature control. At full reactor power (3000 kw), under stagnant conditions (unpressurized), the temperatures recorded for units 1 through 4 were 290°, 390°, 310°, and 200°F, respectively. A small flow of helium into the assembly lowered these temperatures slightly, as did a vacuum. Internal pressurization of the assembly with air produced no change until the external coolant water pressure (approximately 8 psig) was offset. With air pressure of 14 psig, a gradual rise in the respective unit temperatures to 400°, 550°, 450°, and 260°F was observed. Additional pressurization to 18 psig had little effect upon specimen temperatures. The internal air pressure was therefore reduced to 14 psig and maintained for the remainder of the one-month irradiation period. During this period, the average exposure temperatures of the individual units fluctuated no more than ±5°F. The thermal gradients detected in units 1, 2, and 3 did not exceed ±20°F. In unit 4, a temperature spread of ±25°F was noted with a mean temperature of 260°F.

#### POST-IRRADIATION EVALUATION

Decanning of the multitemperature assembly and the testing of specimens were performed at NRL in the Metallurgy Division Hot Cell Facility. The neutron flux monitors incorporated in the assembly were analyzed by two radiation counting laboratories and indicated a total neutron dosage (>1.0 Mev) of  $6.6 \times 10^{18}$  n/cm<sup>2</sup> and  $5.0 \times 10^{18}$  n/cm<sup>2</sup> for the A212B and A302B materials, respectively.

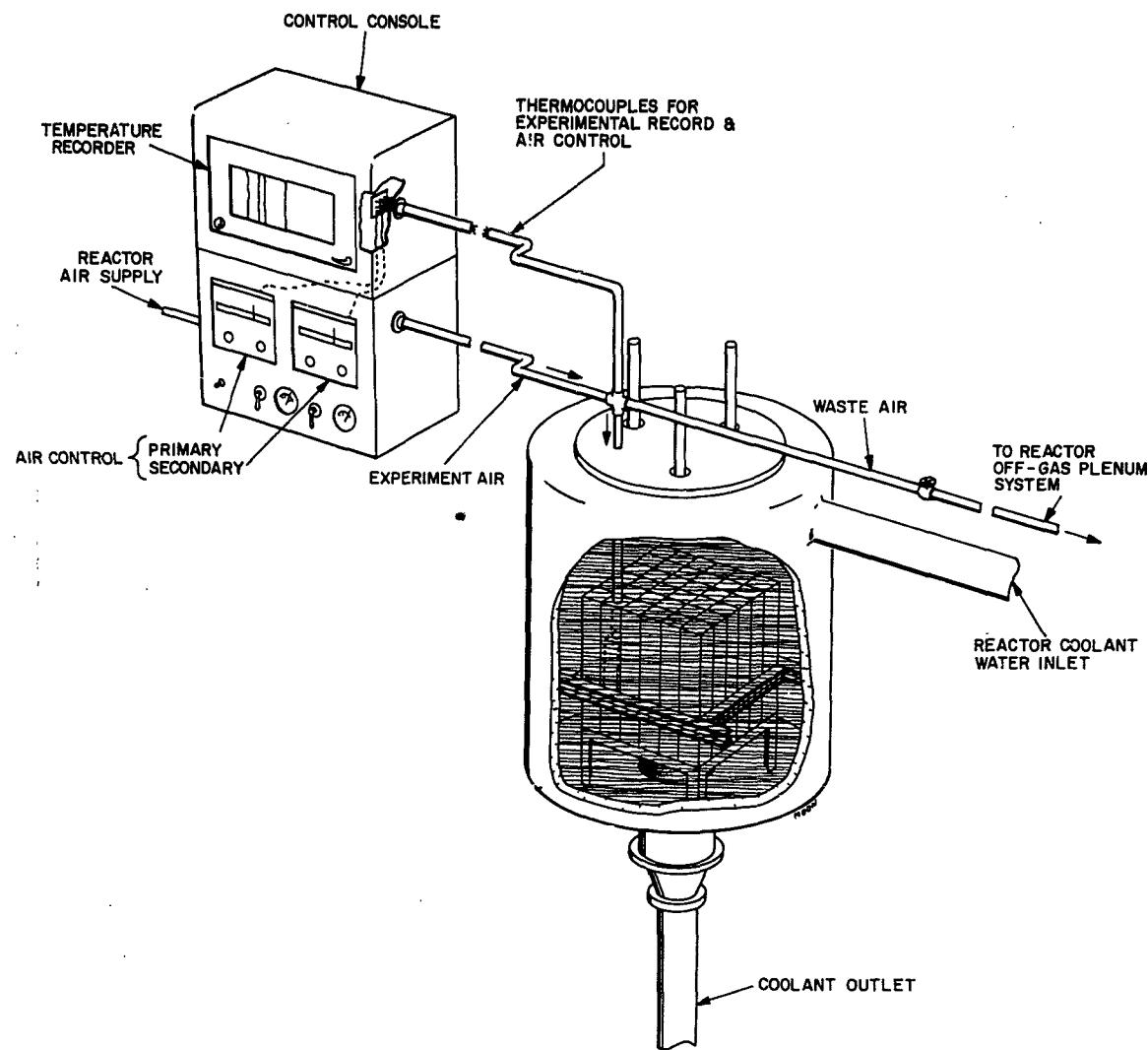


Fig. 4 - Schematic view of multitemperature irradiation experiment,  
showing system for temperature control

The notch ductility exhibited by the 4-in. A212B steel after irradiation at the various temperatures is shown in Fig. 5. Similar results for the 6-in. A302B steel are presented in Fig. 6. For an index of radiation damage, the shift in the transition temperature at an impact energy of 30 ft-lb was used. From these data, it is seen that no appreciable correction of neutron-induced "damage" is effected during irradiation at temperatures of  $450^{\circ}\text{F}$  or less. The amount of shift in both the A212B and A302B materials irradiated at  $400^{\circ}$  and  $450^{\circ}\text{F}$  is only slightly less than that observed for the  $260^{\circ}\text{F}$  exposure. Irradiation of these materials at  $550^{\circ}\text{F}$ , however, produced a significant effect. The transition temperature shifts effected at this temperature are approximately one-hundred degrees less than those produced at the three lower temperatures.

The above data indicate that the process of "self-healing" of neutron-induced effects during irradiation does not occur in a continual stepwise fashion as the irradiation temperature is progressively increased above  $200^{\circ}\text{F}$ . Instead, a critical temperature in the  $450^{\circ}$  to  $550^{\circ}\text{F}$  range must be exceeded for this phenomenon to begin.

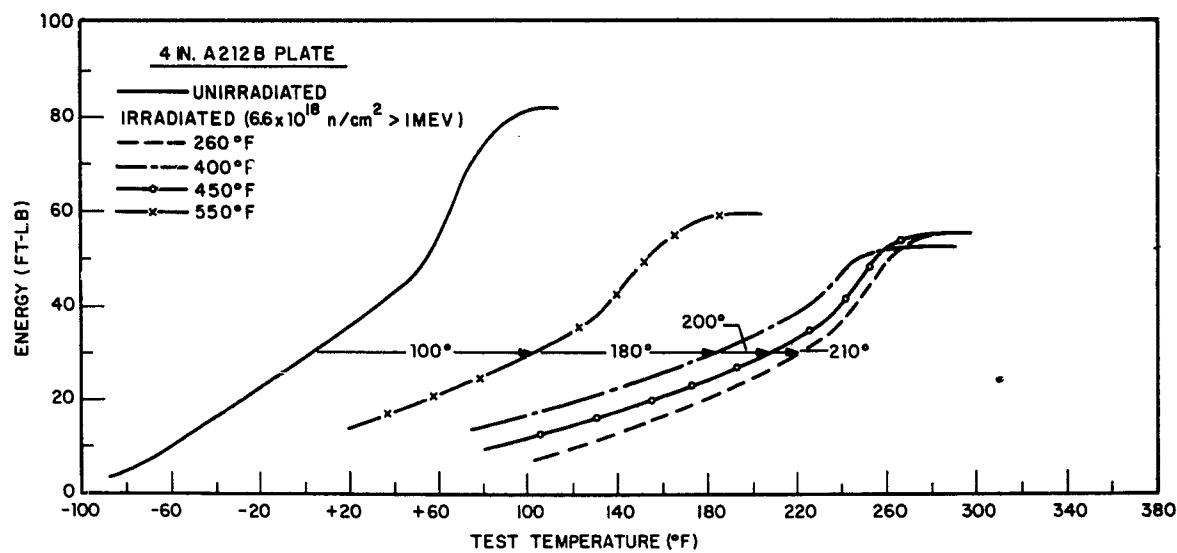


Fig. 5 - Effect of irradiation temperature on notch-ductility properties of 4-in. A212B plate

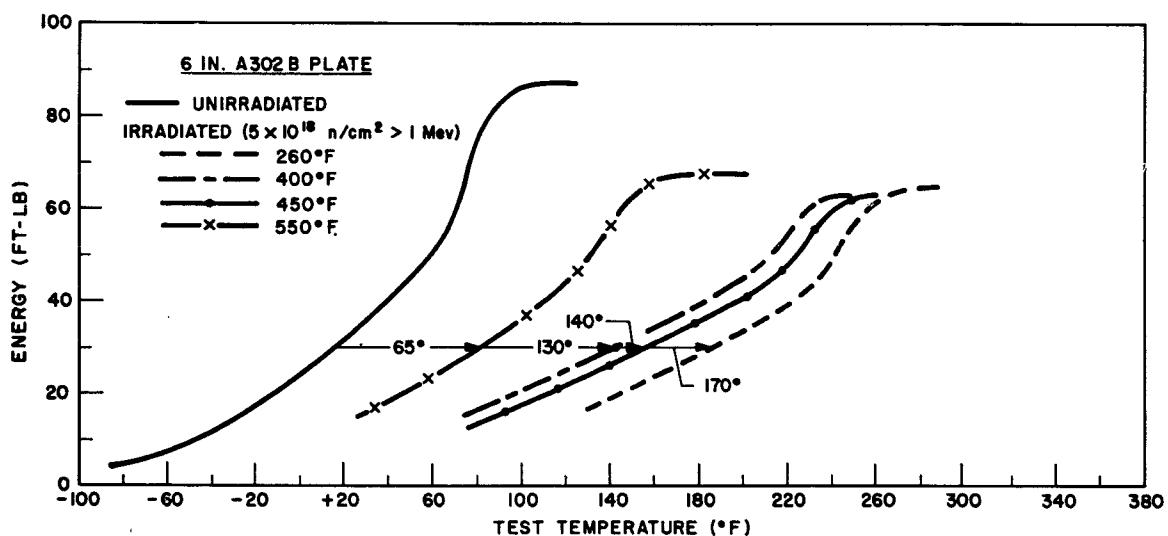


Fig. 6 - Effect of irradiation temperature on notch-ductility properties of 6-in. A302B plate

A comparison of data from this multitemperature assembly with data obtained from previous NRL radiation effects studies on materials irradiated at less than  $200^{\circ}\text{F}$  is shown in Fig. 7. The nineteen points used to establish the basic trend line represent data on four different steel analyses and two weld compositions irradiated in several different nuclear environments. Data from the multitemperature experiment have been superimposed on this graph and are identified according to the exposure temperature adjacent to the material symbol. With the exception of the data for the  $550^{\circ}\text{F}$  irradiation, the points are in good agreement with the basic trend line.

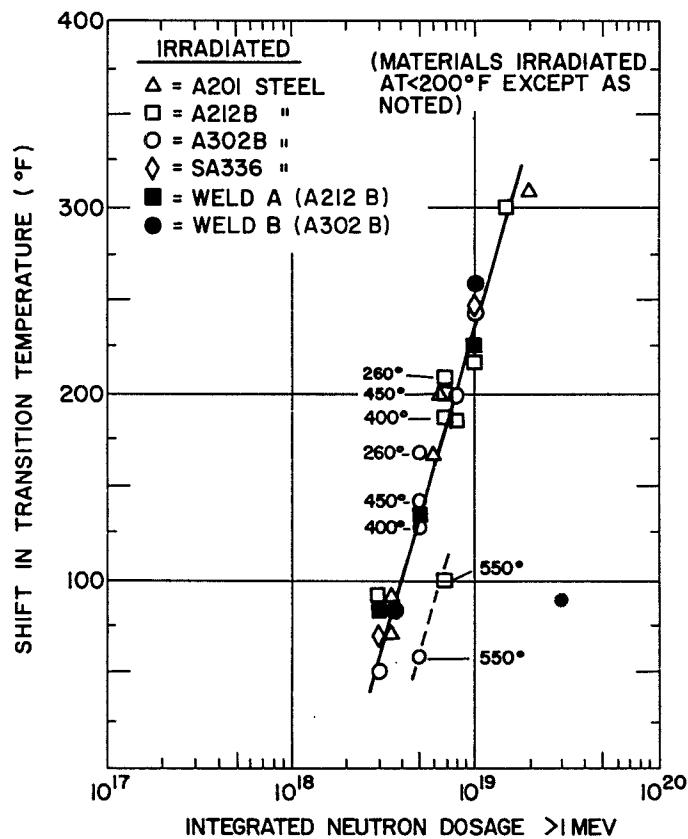


Fig. 7 - Correlation of ductile-to-brittle transition shifts with integrated neutron dosage

Investigations are continuing to define more closely the irradiation temperature at which the process of self-annealing of neutron-induced effects begins in ferritic steels. In addition, experiments are planned to study the behavior of steels irradiated at several elevated temperatures. A series of neutron dosages above  $5 \times 10^{18} \text{ n/cm}^2$  will be utilized to test the  $550^{\circ}\text{F}$  trend line indicated in Fig. 7 and to provide a better understanding of the combined effects of elevated temperatures and neutron irradiation of steel. Studies on the effects of post-irradiation heat treatment on the materials are also included in the planned program.

## SUMMARY AND CONCLUSIONS

An irradiation assembly has been designed for the irradiation of Charpy V-notch specimens at four different temperatures simultaneously in a fuel core facility of the Oak Ridge Low Intensity Test Reactor. A direct comparison of temperature effects during irradiation was therefore possible without the complication of wide variations in the rate or spectrum of incident neutrons.

Evaluation of the notch-ductility properties of a 4-in. A212B and a 6-in. A302B steel plate irradiated at 260°, 400°, 450°, and 550°F indicates that:

1. Shifts in the ductile-to-brittle transition temperature of these materials after irradiation at 400° and 450°F are not significantly different from those observed for the same materials irradiated at 260°F.
2. At an irradiation temperature of 550°F, the process of self-annealing of neutron-induced changes in notch ductility is a concurrent factor, reducing the transition-temperature shift to approximately one-hundred degrees less than that observed for materials irradiated at temperatures less than 450°F.
3. The transition-temperature shifts observed for irradiation temperatures of 260°, 400°, and 450°F are in good agreement with a basic trend established with several different plate and weld metals irradiated in several nuclear environments at temperatures under 200°F.
4. On the basis of the above, it is concluded that no appreciable annealing of radiation effects occur during irradiation at temperatures under 450°F.

## ACKNOWLEDGMENTS

The important contributions of Mr. F. F. Newman in the design and fabrication of the experimental assembly, of Mr. R. F. Bryner in providing the required control instrumentation at the reactor, and of Mr. J. H. Hendricks in the remote evaluation of irradiated specimens are gratefully acknowledged.

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APPENDIX A  
INFORMATION ON THE STEELS

CHEMICAL ANALYSIS

Material No.	Type	Thickness (in.)	Composition (wt-%)							
			C	Mn	Si	P	S	Ni	Cr	Mo
1	Plate (A212B)	4	0.26	0.76	0.24	0.011	0.031	0.22	0.20	0.02
2	Plate (A302B)	6	0.20	1.31	0.25	0.012	0.023	0.20	0.17	0.47

FABRICATION PROCEDURES

Material No. 1

Base Material - Supplied as 4-in.-thick steel plate, conforming to ASTM Specifications A212 Grade B.

Heat Treatment -

1. Austenized at 1650° F for 2 hr, water quenched.
2. Tempered at 1175° F for 1 hr per inch of thickness.

Material No. 2

Base Material - Supplied as 6-in.-thick steel plate, conforming to ASTM Specifications A302 Grade B.

Heat Treatment -

1. Austenized at 1650° F for 2 hr, water quenched.
2. Tempered at 1200° F for 1 hr per inch of thickness.

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<p><b>UNCLASSIFIED</b></p> <p>Naval Research Laboratory. Report 5629. EFFECT OF IRRADIATION TEMPERATURE ON NEUTRON-INDUCED CHANGES IN NOTCH DUCTILITY OF PRESSURE-VESSEL STEELS, by L. E. Steele and J. R. Hawthorne. 9 pp. &amp; figs., June 28, 1961.</p> <p>In an effort to understand more fully the effects of elevated irradiation temperatures upon the shift (increase) in the ductile-to-brittle transition temperature of two pressure-vessel steels, an experimental assembly containing four groups of specimens of these steels has been irradiated in the core of the Oak Ridge Low Intensity Test Reactor. Through the careful design of each unit and by manipulation of the outer experimental containment sheath by air pressure, four different irradiation temperatures, 260°, 400°, 450°, and</p>	<p><b>UNCLASSIFIED</b></p> <p>Naval Research Laboratory. Report 5629. EFFECT OF IRRADIATION TEMPERATURE ON NEUTRON-INDUCED CHANGES IN NOTCH DUCTILITY OF PRESSURE-VESSEL STEELS, by L. E. Steele and J. R. Hawthorne. 9 pp. &amp; figs., June 28, 1961.</p> <p>In an effort to understand more fully the effects of elevated irradiation temperatures upon the shift (increase) in the ductile-to-brittle transition temperature of two pressure-vessel steels, an experimental assembly containing four groups of specimens of these steels has been irradiated in the core of the Oak Ridge Low Intensity Test Reactor. Through the careful design of each unit and by manipulation of the outer experimental containment sheath by air pressure, four different irradiation temperatures, 260°, 400°, 450°, and</p>	<p><b>UNCLASSIFIED</b></p> <p>Naval Research Laboratory. Report 5629. EFFECT OF IRRADIATION TEMPERATURE ON NEUTRON-INDUCED CHANGES IN NOTCH DUCTILITY OF PRESSURE-VESSEL STEELS, by L. E. Steele and J. R. Hawthorne. 9 pp. &amp; figs., June 28, 1961.</p> <p>In an effort to understand more fully the effects of elevated irradiation temperatures upon the shift (increase) in the ductile-to-brittle transition temperature of two pressure-vessel steels, an experimental assembly containing four groups of specimens of these steels has been irradiated in the core of the Oak Ridge Low Intensity Test Reactor. Through the careful design of each unit and by manipulation of the outer experimental containment sheath by air pressure, four different irradiation temperatures, 260°, 400°, 450°, and</p>
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550°F, were maintained for a one-month irradiation period. Post-irradiation evaluation of specimens indicated no significant temperature effect for the materials irradiated at less than 450°F, the shift in the ductile-to-brITTLE transition temperature being about the same for each of the three irradiation temperatures; however, there was a significant effect as a result of irradiation at 550°F, the shift being roughly one-hundred degrees less. The transition-temperature shifts for materials irradiated at less than 450°F were in good agreement with data from nineteen earlier experiments in which the specimen temperatures during irradiation were less than 200°F. The shift increases linearly with the logarithm of neutron flux dosage, with data for irradiation below 450°F falling along a single line; the data for materials irradiated at 550°F fall on a line displaced toward lower transition-temperature shifts.

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